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ADVISORY COUNCIL

SUBCOMMITTEE ON MANUAL OF PROCEDURES

UNIVERSITY OF CALIFORNIA

Wednesday ✓
February 22, 1978
1:00 P.M.

939 Ellis Street
San Francisco,
California

M I N U T E S

COMMITTEE MEMBERS PRESENT: Lowell V. Smith, Chairperson
Shirley J. Campbell, Joseph D.
Coons, Hiram Wolch.

COMMITTEE MEMBERS ABSENT: John L. Nicholson.

STAFF MEMBERS PRESENT: Steve Balestrieri, Dario Levaggi,
Walter Oyung, Meg Sanders.

- - - -

Chairperson Smith called the meeting to order at 1:10 P.M.

APPROVAL OF MINUTES OF FEBRUARY 8, 1978

Mrs. Campbell requested the following corrections:

Page 1, second line from bottom, "TAKEN FROM GAS STREAMS"
to be deleted, and line above it to read "...DETERMINATION
OF AMMONIA IN EFFLUENTS"

Page 2, Lab 1, 3.2 - line 2 (instead of line 1)
5.1 - line 1, to read, "...0.3144 g"
LAB 7A, to read, "...PERMANGANATE PROCEDURE)"
Lab 7A, 3.3 - line 2, to read, "...and dilute to one liter."
5.1 - line 2 (instead of line 1)

Page 3, Lab 7A, 5.1 - line 3, to read, "...Dilute 1:5 to make a
working standard solution ..."
Lab 7B, 4.7 - line 3, to read, "Transfer the solution to..."
(delete "This is characterized by an ammoni odor.")
4.8 - line 3, to read, "...100% transmittion. From the
standard curve..."
5.1 - line 5, to read, "this stock standard solution
1:10 to obtain..."
6 - line 5, to read, "Flask pressure = (760 mm - "

Page 4, Lab 14, 2.1.1-line 5, to read, "...250-300 ml/min air,"

Page 4, Lab 14, 2.1.1 - line 1, to read, "...analytical column is used,..."

4.1 - line 1, to read "...the total volume of the impinger liquid..."

Page 5, Lab 19, 4.3 - line 2, to read "...sulfuric acid to 500 ml of distilled water and dilute to one liter."

Lab 8, 2.1 - line 2, to read "...column which is packed"

Page 6, 5.1 - line 4, to read "...approximately 100 ugm/ml of each phenol."

Page 7, Lab 16, 5.2 - line 4, to read, "...approximately 0.5 L"

5.5 - line 3, to read "...approximately 0.5 L per"

Dr. Wolch moved that the Minutes be approved as corrected; seconded by Mrs. Campbell; carried.

Mr. Coons arrived at 1:30 P.M.

REVIEW OF LABORATORY METHODS INVOLVING METALS

LAB 2A was changed to LAB 2 and LAB 2B was deleted.

Mr. Levaggi made the following corrections for LAB 2:

TITLE: "DETERMINATION OF BERYLLIUM IN EFFLUENTS OR ATMOSPHERIC PARTICULATE MATTER"

3.4 - "Hydrochloric Acid..."

3.5 - line 1, "Sulfuric Acid (12N). Slowly add 33.6 ml..."

4.4 - line 2, to read "sensitivity at a wave length of 234.8 nm by use of beryllium..."

4.4.1 -line 2, to read, "record the percent absorption.
Convert the readings to absorbance units."

4.4.2 -line 3, to read "...hydrochloric acid to be within..."

5.2 - line 1, to read, "...Dilute 5.0 ml of..."

line 2, to read, "...solution to 100.0 ml with..."

The Subcommittee made the following corrections to LAB 2:

4.1 - line 3, to read, "brown fumes are evident, thus
destroying all organic matter. Cool to"

4.3 - line 1, to read, "Prepare a blank filter..."

4.4 - line 4, add, "The blank is used to set the instrument at 0% absorption."

4.4.4- line 1, to read, "Standards should be aspirated after..."

5.2 - line 3, to read, "...a concentration of 5 ugm Be/ml.
At least 3"

5.2 - line 4, to read, "individual standards, normally in the range of 0 to 5 ugm Be/ml, are".

line 6, to read, "with 25% V/V hydrochloric acid."

line 7, to read, "The working standard solution..."

LAB 4A - Mr. Levaggi made the following changes:

2.2 - "Atomic absorption spectrophotometer

2.2.2 - "Air supply"

2.2.3 - "Lead hollow cathode lamp"

2.3 - "Hot plate"

2.4 - "500 ml Phillips beakers"

3.2 - "Dilute Nitric acid. Dilute 10 ml of concentrated Nitric acid to 1 liter with distilled water."

3.3 - "Lead Nitrate ($\text{Pb}(\text{NO}_3)_2$)"

4.1 - line 3, to read, "and place in a 500 ml Phillips beaker."

4.1.3- line 2, to read, "5 ml. Cool to room temperature, and transfer quantitatively to a 50 ml volumetric"

- line 3, to read, "flask using distilled water."

- delete line 4

4.3 - line 2, to read, "sensitivity at a wave length of 283.3 nm by use of lead standards,..."

4.4 - line 2, to read, "record the percent absorption for each. Convert the readings to absorbance units."

4.4.1- line 1, to read, "Samples that have..."

- line 3, to read, "must be diluted with dilute Nitric..."

5.1 - line 1, to read, "Standard stock lead solution. Weigh and transfer to a 1 liter volumetric flask"

- line 2, to read, "1.598 g of lead nitrate. Dissolve and bring to volume with dilute Nitric acid."

- line 3, to read, "This solution contains 1000.00 ugm Pb/ml."

5.2 - line 1, to read, "...At least 3 individual"

- line 2, to read, "standards, normally in the range of 0 to 20 ugm Pb/ml, are freshly prepared by"

- delete lines 4 and 5.

The Subcommittee made the following change for LAB 4A:

- 5.3 - line 1, to read, "Prepare a standard curve on linear graph paper by plotting absorbance of the individual standards vs."
- line 2, to read, " concentration of lead."

Mr. Dale Iverson
Union Oil
Rodeo, California

asked what portion of the filter in LAB 4A, CALCULATION 6. would be used. Mr. Levaggi and Mr. Oyung replied that the usual portion would be 1/4 of the total filter.

LAB 4B - Mr. Levaggi made the following corrections:

- 1. - line 5, to read, "...atomic absorption spectroscopy."
- line 7, to read, "determined by atomic absorption spectroscopy..."
- 2.2- to read, "Atomic absorption spectrophotometer"
- 2.2.2- to read, "Air supply"
- 2.2.3- to read, "Lead hollow cathode lamp"
- 2.3 - to read, "Hot plate"
- 2.4 - to read, "500 ml Phillips beaker"
- 3.1 - to read, "Concentrated Nitric Acid (69 - 71%)"
- 3.2 - line 1, to read, "Dilute Nitric acid. Dilute 10 ml of concentrated nitric acid"
- line 2, to read, "to 1 liter with distilled water."
- 3.3 - to read, "Lead Nitrate ($\text{Pb}(\text{NO}_3)_2$)"
- 4.1.2 -line 2, to read, "...and cover the beaker with a"
- 4.1.3 -line 4, to read, "Filter each wash, adding each..."
- 4.1.4 - line 2, to read, "5 ml. Cool to room temperature, transfer the filtrate..."
- 4.1.4 -line 3, delete "Allow the sample to cool and make"
- line 4, delete "up to volume with distilled water."
- 4.2 -to read, "From each batch of purchased glass wool analyze a representative portion in the same manner as the samples."

- 4.3 - to read, "Set spectrophotometer parameters to obtain maximum sensitivity of a wavelength of 283.3 nm by use of lead standards, and manipulation..."
- 4.4 - line 2, add, "Convert the readings to absorbance units."
- 4.4.1- line 3, to read, "must be diluted with dilute Nitric..."
- 6.2 - line one to read,"

$$\begin{array}{lcl} \text{*Total mg} & & \text{ugm Pb/ml from Std Curve x 50 x 20} \\ \text{per} & = & \\ \text{Impinger} & & \text{1000 x Vol. of Impinger (ml)} \end{array}$$

LAB 4B - The Subcommittee made the following changes:

- 4.4.3- line 1, to read, "Standards should be aspirated after every fourth"
- 4.5.1- line 2, to read, "monochloride solutions remaining in the impingers."
- 5.1 - line 1, to read, "Weigh standard stock lead solution in a 1 liter volumetric"
- 5.2 - line 1, to read, "Working standard lead solutions. At least 3 individual"
- line 2, to read, "standards, normally in the range of 0-20 ugm Pb/ml, are freshly prepared by"
- delete lines 4 and 5
- 5.3 - line 1, to read, "...standard curve on linear graph paper by plotting absorbance of the individual standards vs."

LAB 6 - Mr. Levaggi reported that LAB 6 is to be replaced with the corresponding EPA procedure.

LAB 9 - Mr. Levaggi discussed the procedure and made the following changes:

- 1. - line 5, to read, "...whereas coatings must be steam"
- line 7, to read, "identification."
- line 10, to read, "...of the different organic classes."
- paragraph 4, line 2, to read, "...and subtractive techniques. First, obtain a chromatogram of the solvent diluted with carbon disulfide."
- paragraph 4, line 5, to read, "and oxygenated hydrocarbons, sulfonation acid is utilized."
- paragraph 4, line 6, delete line

- 2.1 - to read, "Fluorescent indicator analysis apparatus,
as shown in Figure 2
- 2.2 - Pressurized gas manifold, equipped with a Mercury
manometer (Nitrogen or Air)
- 2.6 - to read, "Separatory funnel, 500 ml"
- 2.7 - line 1, to read, "...58 mm diameter x 10 mm high"
- 2.8 - to read, "Gas chromatographic unit. The unit is
equipped with a compatible recorder and flame
ionization detector."
- 3.1 - line 1, to read, "...Grade 923. This is available from"
- line 2, to read, "...Maryland (Davison Code No. 923-08-
- line 3, to read, "08220). Before use, dry the gel at
175°C for 3 hours; transfer"
- 3.2 - line 1, to read, "Fluorescent indicator dyed gel.
This is available from U.O.P."
- line 2, to read, "...Des Plaines, Ill. 60016 (U.O.P."
- line 3, to read, "Product No. 675).."
- 3.3 - to read, "Isopropyl Alcohol"
- 3.4 - to read, "Pressurized Gas, cylinder nitrogen or air"
- 3.5.1- to read, "14' x 1/8" 10% Carbowax 20M/Chrom W, AW
DCMS, 80-100 mesh."
- 3.5.2- line 2, to read, "Chromosorb W, 60-80 mesh."
- 3.5.3- line 1, to read, "15' x 1/8" 10% N₂ N₂ Bis 2 cyano-ethyl..."
- line 2, to read, "Chromosorb W, 60-80 mesh."
- 3.8 - to read, "Carbon Disulfide"
- 3.9.1- to read, "Equal volumes of isooctane, heptene and
xylene, or appropriate substitutes. Prepare prior to use."
- 3.9.2- to read, "Equal volumes of isooctane, methyl ethyl
ketone and xylene, or appropriate substitutes. Prepare
prior to use."
- 3.11 - line 1, to read, "...Add the contents of a 1 lb (454 gm)
- line 2, to read, "bottle of..."
- 4.1.1- line 1, to read, "Weigh accurately 0.4 to 0.6 g ($\pm$.1 g)
of coating..."

- 4.1.1 - line 2, to read, "...105 $\pm$ 1° C for three
- 4.1.1 - line 4, to read, "...Results should agree to within $\pm$ 5% of the mean."
- 4.3.1 - line 3, to read, "the weight of coating ($\pm$ 0.1 g)..."
- 4.3.2 - line 2, to read, "70° to 80° C..."
- 4.3.3 - line 1, to read, "...from the water layer by"
- line 2, to read, "...funnel. Record ($\pm$ 1 ml) the volumes of both fractions,"
- line 3, to read, "as well as the weight of..."
- line 4, to read, "oxygenates are not present, the..."
- line 5, to read, "...determined by 4.2.4."
- line 6, to read, "The water layer is discarded. If..."
- line 10, to read, "described in 4.1.1. The combined..."
- 4.3.4 - line 1, to read, "For further processing,..."
- 4.4 - line 1, to read, "...for Solvents and"
- line 2, to read, "Hydrocarbon Fractions of Distillates not containing Oxygenated"
- line 3, to read, "Hydrocarbons."
- 4.4.1 - line 1, to read, "...suspend the column. While"
- line 2, to read, "...its entire length, add small..."
- line 5, to read, "Dyed Gel. Start the..."
- 4.4.3 - line 2, to read, "connect the column to the gas manifold. Apply 40 to 80 mm Hg"
- 4.4.4 - line 3, to read, "...and avoid handling the column..."
- 4.5 - line 10, to read,
- $$\% \text{ V/V Olefins} = \frac{L_o}{L_t} \times 100$$
- 4.5 - line 14, to read, "...chromatography (4.8) for..."
- line 15, to read, "pound identification."
- 4.6 - line 1, to read, "Modified FIA for Solvents and Hydrocarbon Fractions"
- line 2, to read, "of Distillates Containing Oxygenated Hydrocarbons"

- 4.6.1 -line 2, to read, "...its entire length, add small..."
- line 4, to read, "...layer of Fluorescent Indicator"
- line 5, to read, "Dyed Gel and 3 drops..."
- line 6, to read, "While adding..."
- 4.6.5- line 1, page 9, to read "...When the sample has..."
- line 3, page 9, to read, "...relative zone lengths..."
- 4.6.6- line 1, to read, "A standard solution prepared as in 3.9.2 should be run"
- 4.7 - line 4, to read, " $L_t = L_s + L_a + L_{ox}$ "
- line 8, to read, " L_{ox} = Total length of the oxygenate zone"
- line 11, to read, "% V/V Oxygenated Hydrocarbon = $\frac{L_{ox}}{L_t} \times 100$ "
- 4.7.1--line 3, to read, "...if indefinite, continue..."
- line 4, to read, "...compound identification."
- 4.8.1.1- ~~deleted~~
- 4.8.1.2- change to 4.8.1.1
- to read, "Columns described in 3.5.1 and 3.5.2 are not recommended where high boiling oxygenates are present or suspected."
- 4.8.1.3- change to 4.8.1.2
- 4.8.2- to read, "Prepare a 2 to 5% v/v (± 1 ml) solution..."
- 4.8.3- line 5, to read, "peaks and complete masking of some..."
- 4.8.4- line 1, to read, "...transfer 2 to 3 ml of the"
- line 4, to read, "...mixer for 30 to 40 seconds. Let the sample..."
- 4.9 - line 4, to read, "be made of the sample chromatograms..."
- 4.9.1- line 3, to read, "...4.8.3 identifies"
- line 4, to read, "oxygenated compounds. The..."
- line 8, to read, "...(Dil. Factor * 100)

- 4.9.1- line 9, to read, "*as found in 4.8.2"
- 4.9.2- page 13, line 1, to read, "Total % v/v..."
- line 2, to read, "... (Dil. Factor *) (100)"
 - line 3, to read, "*as found in 4.8.2"
- 4.10 - line 1, to read, "Compliance with..."
- line 3, to read, "...in 4.9.1 and 4.9.2..."
- 4.11 - line 3, to read, "...and quantify the miscible..."
- 4.11.3-line 1, to read, "A standard containing the normally..."
- line 2, to read, "oxygenates is prepared in distilled water (ul/ml). Inject 2.0 ul of"
- line 3, to read, "the standard and record the..."
- line 4, to read, "Retain the chromatogram for future reference."
- 4.12.1-line 2, to read, "... (Total Vol. of Samp. *)
- line 3, to read, "*as found in 4.3.3"

LAB 9, the Subcommittee made the following corrections:

1. - Paragraph 3, line 8, to read, "...determined on the basis"
 - Paragraph 3, line 9, to read, "of the appropriate FIA;
if compliance is not defined sufficiently, gas"
 - Paragraph 3, line 10, to read, "chromatography is necessary..."
- 3.11 - line 6, to read, "mixture into ground glass stoppered
bottles..."
- 4.1.1 - line 3, to read, "...and weigh. Analyze samples"
- 4.5 - line 1, to read, "...calculate v/v"
- line 2, to read, "of each hydrocarbon type to the
nearest 0.1%, as follows:"
- 4.6.2- line 2, to read, "Chill the sample and the hypodermic syringe to 35 to 40° F."
- line 3, to read, "Inject 0.75 ml of the sample 30 mm"
- 4.6.3- line 2, to read, "...a pressure of 40 to 80 mm Hg."
- line 3, to read, "Higher pressure can result"
- 4.6.4- line 2, to read, "carefully wipe the side..."
- 4.8.6- line 3, to read, "...contain C₆ to C₁₂ aliphatics,"

- 4.8.6- line 1, Page 12, to read, "C₇ to C₁₂ aromatics,..."
- line 2, Page 12, to read, "...prepared on a v/v basis"
 - line 3, Page 12, to read, "(ul/ml for ready...."
 - line 4, Page 12, to read, "by the regulation).
- 4.9.1 - line 7, to read, "Total % v/v of each..."
- 4.10 - line 5, to read, "...steam distilled and contain"

Mr. Levaggi said the Figures for LAB 9 will be made more readable.

TIME AND PLACE OF NEXT MEETING

Wednesday, March 29, 1978, 1:00 PM, 939 Ellis Street, San Francisco, California.

The meeting was adjourned at 4:00 PM.

Respectfully submitted,

Mabel Harder
Secretary

ms

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